

Fatigue damage evolution in ZrO_2 -reinforced Al7075 metal matrix composites: from S-N curves to Paris multilinear regimes

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Citation: Vantadori, S., Iacoviello, F., Bellini, C., Di Cocco, V., Ronchei, C., Scorza, D., Zanichelli, A., Fiorillo, R., Fatigue damage evolution in ZrO_2 -reinforced Al7075 metal matrix composites: from S-N curves to Paris multilinear regimes, *Fracture and Structural Integrity*, 78 (2026) 432-453.

Received: 04.08.2026

Accepted: 13.09.2026

Published: 17.09.2026

Issue: 10.2026

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ABSTRACT. Aluminum Matrix Composites (AMCs) are increasingly used in aerospace, automotive and marine applications, where structural components are often subjected to cyclic and time-varying loading conditions. A reliable assessment of their fatigue response is therefore essential for safe design and damage-tolerant applications. In this study, the fatigue behaviour of an Al7075 alloy reinforced with 1.5 wt% zirconium dioxide nanoparticles (ZrO_2) is experimentally investigated, with particular attention to both fatigue lifetime and crack propagation response. The experimental programme first includes monotonic tensile tests, aimed at determining the static mechanical properties



of the investigated nano-reinforced AMC and defining suitable load levels for subsequent cyclic tests. Fatigue life tests are then carried out under cyclic tensile loading at a fatigue ratio of 0.10, while fatigue crack propagation tests are performed at different fatigue ratios, namely 0.10, 0.50 and 0.75. To support the mechanical interpretation of the results, fracture surfaces are examined through optical macrographs and high-resolution scanning electron microscopy, allowing the main crack propagation and failure mechanisms to be identified. The experimental findings are compared with literature data available for both unreinforced aluminum alloys and ZrO_2 -reinforced AMC. The main novelty of the study lies in the integrated analysis of fatigue life, crack propagation behaviour and fractographic evidence within the same experimental framework. To the best of the authors' knowledge, such a comprehensive investigation has not yet been reported in the literature for the composite system examined herein. While previous investigations often considered S-N fatigue response and fatigue crack propagation separately, the present work bridges these two aspects, providing a more complete description of damage evolution in ZrO_2 -reinforced Al7075 AMCs. This integrated dataset offers useful experimental support for the calibration and validation of fatigue damage models for nano-reinforced AMCs operating under service-like cyclic loading conditions.

KEYWORDS. Al7075, Crack propagation, Fatigue life, Nanocomposite, ZrO_2 .

INTRODUCTION

Metal matrix composites (MMCs) are engineered materials composed of two or more constituent phases, primarily consisting of a ductile metallic matrix - typically aluminum, magnesium, titanium, or steel - and at least one reinforcement phase, generally a harder metallic or non-metallic material in the form of continuous or short fibers, particles, or whiskers [1]. As a matter of fact, traditional metallic materials, despite their good mechanical properties, may exhibit limitations in demanding operating conditions involving high temperatures, wear, and corrosive environments [2,3]. The incorporation of reinforcing phases can significantly enhance the strength-to-weight ratio, wear and corrosion resistance, thermal stability, and tailoring capability of the resulting composites [4–8], making MMCs highly attractive for applications in sectors such as aerospace, automotive, electronics, and defence [9,10].

The interaction between the matrix and the reinforcement plays a crucial role in determining the final properties of MMCs [11,12]. Therefore, the fabrication route is of primary importance. MMCs can be produced through liquid-state processes, such as stir casting, or solid-state processes, such as powder metallurgy. In both cases, achieving a uniform reinforcement distribution while limiting particle clustering is essential to ensure consistent mechanical properties [13].

Among MMCs, Aluminum Matrix Composites (AMCs) are widely employed in the aforementioned sectors, where weight reduction without compromising strength and durability is essential [14]. In such a context, significant progress has been made in incorporating nanoparticles, like carbides, oxides and nitrides (with a size less than 100nm), into aluminum alloys, enhancing fatigue strength, wear resistance and hardness [14]. Examples of carbides include SiC (silicon carbide), B_4C (boron carbide) and TiC (titanium carbide) [5,6,11], while oxides include Al_2O_3 (aluminum oxide), ZrO_2 (zirconium oxide), and TiO_2 (titanium oxide) [6,12,14,15]. Nitrides often employed include Si_3N_4 (silicon nitride), TiN (titanium nitride) and AlN (aluminum nitride) [16].

In the recent years, several studies have been carried out on the effect of nanoparticle addition in aluminum matrices [5,6,14–16]. For instance, the tensile strength, hardness and wear resistance of a hybrid AMC made of an Al7075 matrix reinforced with SiC nanoparticles and neem leaf powder were investigated by Hatti et al. [5]. They observed an increase in hardness and a general improvement in the composite mechanical properties, which they attributed to the strong interfacial bond between the matrix and the uniformly dispersed particles. Prakash et al. [6] studied a hybrid AMC consisting of an Al7075 matrix reinforced with different weight fractions of B_4C and Al_2O_3 nanoparticles, produced through the stir casting process.



They found that, for the composite reinforced with 4.5% B₄C and 2% Al₂O₃, the addition of the nanoparticles, along with heat treatment, led to improvements of approximately 3%, 17%, and 10% in tensile strength, hardness, and wear resistance, respectively, compared to composites with lower reinforcement content. The physical, mechanical and tribological properties of an Al6063 matrix reinforced with silicon nitride powder (ranging from 0 to 10 wt%) were, instead, analysed by Kumar et al. [16]. According to their observations, the microstructure exhibited a uniform distribution of particulates in the matrix and the hardness increased with the filler content as well as the tensile strength. Also, the resistance to wear was significantly higher than that of the base aluminum alloy exhibiting excellent tribological characteristics.

Due to the time-varying in-service loading conditions characterising the sectors of application, AMC components are prone to damage accumulation, fatigue crack initiation and propagation, and ultimately sudden fatigue failure [17,18]. Therefore, the study of the fatigue behaviour of AMCs has attracted the attention of many researchers in recent years, as reported in the interesting review by Malaki et al. [19].

In particular, both micro-sized [20-22] and nano-sized reinforcements [23,24] have been incorporated into aluminum alloy matrix in order to improve its fatigue resistance. However, it has been demonstrated that, compared to micro-sized particles, the AMCs with nano-sized particles (with sizes less than 100nm) exhibit greater fatigue performance [19,25,26]. For instance, Senthilkumar et al. [25] investigated the low-cycle fatigue behaviour of an Al2014 matrix reinforced with either 10wt% micro-sized Al₂O₃ particles or a hybrid reinforcement consisting of 8 wt% micro-sized and 2 wt% nano-sized Al₂O₃ particles, fabricated through hot extrusion. Low cycle fatigue tests were performed under strain-controlled, pull-push cyclic loading conditions, primarily at a strain ratio of -1. The hybrid composite exhibited a higher fatigue life when compared to the one with only micro-sized particles. This behaviour was attributed to the nano-sized reinforcement more effectively restricting dislocation mobility through the Orowan strengthening mechanism, in which dislocations bow around the closely spaced nanoparticles [14]. Moreover, the improved fatigue performance of nano-sized reinforcing AMCs compared to macro-sized ones can be attributed to the exceptionally high aspect ratio of the former, marked by a large surface area compared to their volume, which promotes extensive interfacial interaction between the matrix and the reinforcement [19]. Different nanoparticles, such as TiO₂, ZrB₂, SiC, Si₃N₄, and TiB₂, have been added to the aluminum alloy matrix in order to investigate their influence on the fatigue behaviour. Divagar et al. [27] investigated the fatigue strength of AMCs produced via the stir casting process. The research involved reinforcing the base metal, represented by an Al7075-T651 matrix, with varying volume fractions of SiC (5%, 10%, and 15%) and a constant 5% of Al₂O₃ nanoparticles. Rotary bending fatigue tests were performed at different stress levels. The results showed that fatigue strength increased with increasing SiC particle percentage up to 10%, with the composite containing 10% SiC and 5% Al₂O₃ exhibiting a 12% higher fatigue strength than the base metal and other investigated nanocomposites. Jaber et al. [28] experimentally analysed the electrical conductivity, magnetic, and fatigue properties of an Al6063-T4 matrix reinforced with 3, 5, and 7 wt% of TiO₂ nanoparticles. Fatigue behaviour was assessed using rotating bending fatigue tests. The nanocomposite containing 7 wt% TiO₂ demonstrated enhanced fatigue life and strength, showing a 13% increase in fatigue strength compared to the base metal. Zhang et al. [29] performed cyclic tension-compression tests, under a fatigue ratio of -1, on an Al6111 matrix reinforced with 1, 2, and 3 vol% of ZrB₂ nanoparticles. The nanocomposites showed higher fatigue limits than the unreinforced alloy, with the 2 vol% ZrB₂ nanocomposite achieving the highest fatigue limit of 125MPa, representing a 31.5% increase compared to the base alloy. Agrawal and Srivastava [23] investigated the fatigue behaviour of an Al7075 matrix reinforced with Si₃N₄ and TiB₂ particles, fabricated via stir casting. Various compositions were investigated, including 4 wt% Si₃N₄ with 2, 4, or 6 wt% TiB₂. High-cycle fatigue tests were performed under constant-amplitude cyclic tensile loading with a fatigue ratio of 0.1. The addition of reinforcement particles significantly improved the fatigue performance and crack growth resistance of the composites, with the nanocomposite containing 4 wt% Si₃N₄ and 6 wt% TiB₂ exhibiting the longest fatigue life, corresponding to a 128% increase compared to the base alloy.

Since the research in the field of AMCs fatigue is still open, the goal of the present paper is to thoroughly investigate the fatigue behaviour of Al7075 matrix reinforced with zirconium dioxide nanoparticles (1.5 wt% ZrO₂). To this aim, an experimental campaign is carried out comprising preliminary monotonic tensile tests, performed according to the ASTM E8/E8M-22 standard [30], in order to determine the static mechanical properties of the investigated AMC and to define the reference load levels for the subsequent cyclic tests. Cyclic tensile tests are then performed at a fatigue ratio of 0.10, in accordance with the ASTM E466-21 standard [31] for S-N curve determination. Finally, fatigue crack propagation tests are conducted under different fatigue ratios (0.10, 0.50, and 0.75), according to the ASTM E647-23a standard [32] for the determination of fatigue crack propagation rate curves.

Moreover, for the specimens tested to determine the S-N curves, the fracture surfaces are examined through high-resolution Scanning Electron Microscope (SEM) images in order to identify the preferential fatigue crack-initiation sites and the main features associated with fatigue failure. In addition, for the specimens tested in the fatigue crack propagation tests, the



micromechanisms involved are investigated through detailed fractographic analysis of the fracture surfaces, based on both optical macrographs and high-resolution SEM images, corresponding to different Stress-Intensity Factor (SIF) range levels. Early results from this campaign are reported in Ref. [33]; however, the present study provides a substantially expanded experimental dataset. The key innovation of this study lies in the simultaneous investigation of fatigue life and fatigue crack propagation behaviour, an approach that has rarely been addressed in the existing literature. To the best of the authors' knowledge, no previous study on ZrO₂-reinforced Al7075 metal matrix composites has reported both experimental S–N fatigue curves and crack propagation data within the same integrated experimental program. By combining lifetime estimation with crack propagation analysis within a unified experimental framework, a more comprehensive understanding of damage evolution under cyclic loading is achieved. This dual-focus methodology enhances the interpretation of material behaviour and provides unique experimental data for the calibration and validation of numerical fatigue models, particularly in the context of nano-reinforced aluminum matrix composites operating under variable service conditions.

The paper is, hence, structured as follows. Section 2 is devoted to the AMC characterisation - by means of both a chemical and a microstructural analysis - and to the description of the experimental static and fatigue tests performed on the AMC. Section 3 deals with the discussion of the obtained results in terms of static tensile curves, S–N curve, and fatigue crack propagation rate curves, together with optical macrographs and SEM images of the fracture surfaces. In Section 4, the results are compared with data available in the literature, while the main conclusions are summarised in Section 5.

MATERIAL CHARACTERISATION AND TESTING METHODS

Material characterisation

The nanocomposite investigated in this study is a commercial AMC based on an Al7075 alloy, reinforced with 1.5 wt% ZrO₂ nanoparticles with an average particle size of 40nm.

Its chemical composition, determined by using an optical emission spectrometer, is shown in Tab. 1, being dominated by aluminum (89.23 wt%), with significant additions of zinc (5.50 wt%) and magnesium (2.40 wt%), consistent with 7xxx-series aluminum alloys known for high strength through precipitation hardening. Copper (1.22 wt%) further enhances mechanical performance. Minor elements such as iron, silicon, manganese, and chromium contribute to improving castability, corrosion resistance, and microstructural stability. Titanium (0.10 wt%) is added for grain refinement. The overall zirconium content of 0.15 wt% supports thermal stability and resistance to recrystallization.

Zn	Mg	Cu	Fe	Si	Mn	Cr	Zr	Ti	Al
5.50	2.40	1.22	0.50	0.40	0.30	0.20	0.15	0.10	Bal.

Table 1: Chemical composition of the examined AMC, expressed as weight percentage.

The AMC microstructure is analysed by using an optical microscope. The sample is prepared for the microstructure analysis following the standard procedure. After cutting the sample from a plate through an abrasive disk saw, it is mounted in phenolic resin and ground through a grinder equipped with a silicon carbide disk. This operation is carried out in several steps using abrasive papers with grit sizes ranging from P320 to P1200. Afterwards, the sample is polished using a diamond aqueous suspension applied with a felt disk. To reveal the microstructure, the sample is etched according to the ASTM E407-07(2015)e1 standard [34]. In particular, the etchant number 6 is used, with the sample immersed in a heated solution of nitric acid and water and rinsed in cold water after 40s.

In addition, SEM–EDX analysis is performed on a polished and etched sample to investigate the presence of non-metallic inclusions and possible ZrO₂ nanoparticle agglomerates. The sample is prepared following the same metallographic procedure adopted for optical microscopy, including cutting, mounting in phenolic resin, grinding with silicon carbide abrasive papers from P320 to P1200, polishing with an aqueous diamond suspension and etching in accordance with ASTM E407-07(2015)e1 [34]. The polished surface is then examined by scanning electron microscopy, while energy-dispersive X-ray spectroscopy is used to determine the elemental composition of the observed particles.

As shown in Fig. 1(a), the grains exhibit a layered or banded structure resulting from the lamination process applied during plate manufacturing. Indeed, the plastic deformation induced by the rolling process elongates the grains along the rolling direction, resulting in an average grain size of approximately 250 μm in the rolling direction and about 25-50 μm in the through-thickness direction. Grain size is quantitatively evaluated using the linear intercept method in accordance with the ASTM E112-13(2021) standard [35], measuring a statistically significant number of grains along both directions. The

pronounced grain elongation suggests the development of a typical rolling-induced texture, which is expected to contribute to anisotropy in the mechanical response of the material.

SEM-EDX analysis (Fig. 1(b)) reveals the presence of non-metallic SiO_2 inclusions, whereas no agglomerates of ZrO_2 nanoparticles are observed.

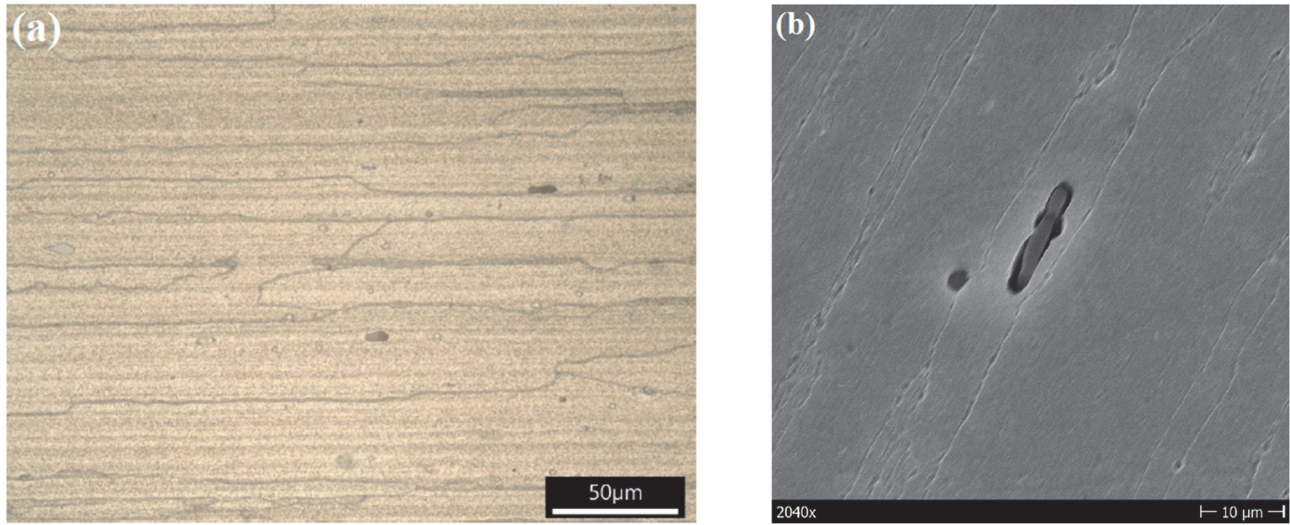


Figure 1: Microstructural characterisation of the AMC: (a) optical micrograph showing the grain structure; (b) SEM image revealing non-metallic SiO_2 inclusions.

Static tensile tests

The quasi-static mechanical properties of the AMC are determined through tensile tests, according to the ASTM E8/E8M-22 standard [30]. The specimens are machined from a plate having a thickness of 10mm. The sizes of the specimens are shown in Fig. 2.

A universal testing machine equipped with a 100kN load cell is used to perform the tests. Each specimen is secured in the machine wedge grips, and the testing apparatus is zeroed to avoid undesired preloads. The crosshead speed is set to 1.2 mm/min, corresponding to an apparent strain rate of 0.015 min^{-1} . Three specimens are tested.

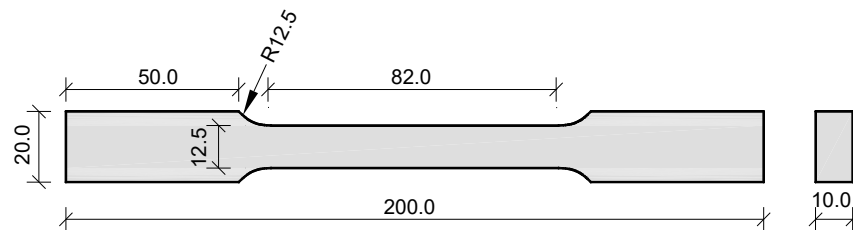


Figure 2: Tensile tests: geometry of the AMC specimen (sizes in mm).

Cyclic tensile tests

The fatigue behaviour of the AMC is investigated through force-controlled constant amplitude axial fatigue tests, according to the ASTM E466-21 standard [31]. The specimens are machined from the aforementioned plate; however, in this case, the thickness is reduced to 4mm to comply with the standard specimen dimensions. The sizes of the specimens are reported in Fig. 3.

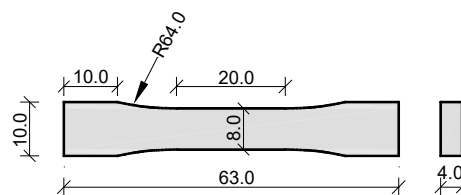


Figure 3: Cyclic tensile tests: geometry of the AMC specimens (sizes in mm).

For each test, the specimen heads are secured on the testing machine through special jaws that prevent slippage.

A cyclic axial tensile load is applied, being characterised by a sinusoidal shape, with a frequency of 80 Hz, and a fatigue ratio $R = 0.10$. Ten stress levels are selected, defined in terms of maximum stress as percentages of the ultimate tensile strength (from 55% to 6%). Three fatigue tests are conducted at each stress level. Each test is terminated either upon specimen failure or when the number of loading cycles reaches $2(10)^6$. However, if a run-out is observed, no further tests are performed at that stress level, and testing proceeds to the next lower stress level.

Fatigue crack propagation tests

The fatigue crack propagation behaviour of the AMC is evaluated in accordance with ASTM E647-23a [32], allowing the fatigue crack propagation rate curve to be determined, as well as the threshold stress intensity factor range, ΔK_{th} , using the load-shedding technique.

The samples used are standard C(I) specimens, whose sizes are reported in Fig. 4. The specimens are machined from the same metal plate used for the tensile samples, with a thickness of 10 mm.

For these tests, the same testing machine used for the cyclic tensile tests is employed, but in this case, it is equipped with pins specifically designed for the aforementioned specimens. For each test, the specimen is secured on the testing machine through pins inserted in the specimen holes, and a clip gauge is mounted on the notch mouth, in order to measure the crack length throughout the experiment using the compliance method.

Constant amplitude sinusoidal loading is considered, with a frequency of 30Hz and three different values of the fatigue ratio, R , that is: 0.10, 0.50 and 0.75. Each specimen is precracked before testing to remove the effect of the machined notch and obtain a sharpened crack.

The fatigue crack propagation rate curves are obtained using three specimens for each R value. For the determination of ΔK_{th} , the load-decreasing parameter, used in the load-shedding procedure, is set to -0.15 mm^{-1} , with two specimens tested under the same conditions.

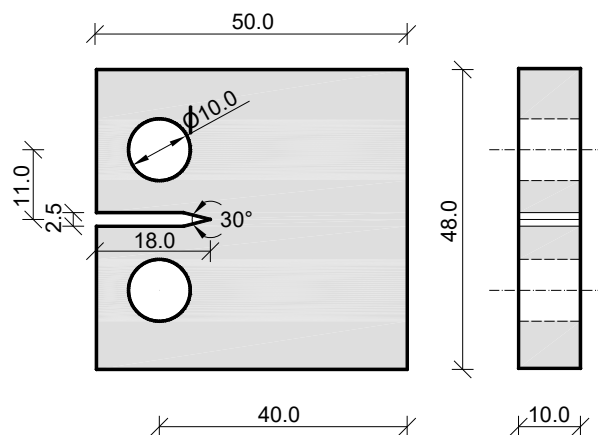


Figure 4: Fatigue crack propagation tests: geometry of AMC specimens (sizes in mm).

RESULTS AND DISCUSSION

Tensile behaviour

The results of the tensile tests are shown in Fig. 5, where stress is calculated based on the actual cross-sectional area of the specimen, and apparent strain is derived from the crosshead displacement. The toe region, located at the beginning of the curve and caused by specimen seating, is adjusted by extending the linear region to the zero-stress axis. The intersection point is considered the correct zero-strain point, and all the apparent strains are measured from that point.

The obtained stress-strain curves exhibit the typical behaviour of a ductile material, with an initial linear elastic region followed by yielding, strain hardening, necking, and, finally, fracture. The yield strength is determined by using the offset method by considering an offset of 0.2%. Therefore, the yielding stress, $\sigma_{y0.2\%}$, is equal to $521 \pm 1 \text{ MPa}$, corresponding to an elastic apparent strain of $3.87 \pm 0.03\%$. The ultimate tensile strength, σ_u , is equal to $584 \pm 1 \text{ MPa}$, while the plastic apparent strain is equal to $11.97 \pm 0.37\%$. The fracture strength, σ_f , is equal to $557 \pm 4 \text{ MPa}$.

The obtained results exhibit minimal scatter, with the coefficients of variation calculated for $\sigma_{y0.2\%}$, σ_u and σ_f equal to 0.19%, 0.17%, and 0.72%, respectively.

Fig. 6 presents a macrograph of the fracture surface, characterised by a V-shaped morphology. The fracture location is close to the centre of the gauge length.

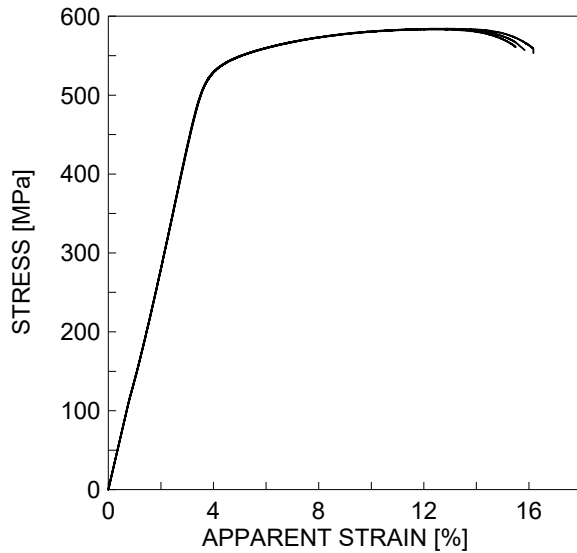


Figure 5: Stress-strain curves under tensile loading.

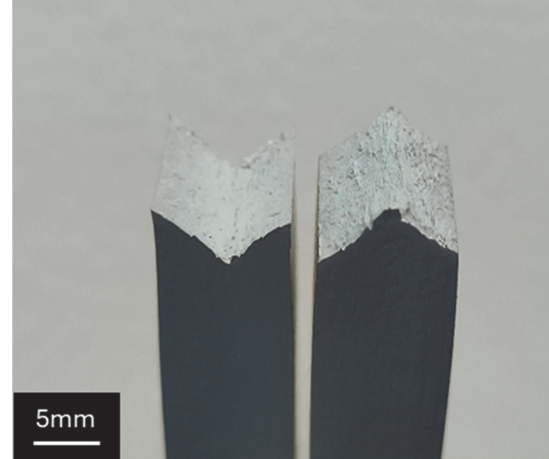


Figure 6: Example of the fracture surface of a tested specimen under tensile loading.

S-N curve

The cyclic tensile test results, in terms of applied stress amplitude, σ_a , and number of loading cycles to failure, N_f , are presented in Tab. 2.

SPECIMEN No.	σ_a [MPa]	N_f [Cycles]
1	145	14302
2	145	18226
3	145	12370
4	65	48455
5	65	31010
6	65	40572
7	50	61206
8	50	81206
9	50	78336
10	40	46480
11	40	76480
12	40	48365
13	35	373685
14	35	350000
15	35	286420
16	29	1418755
17	29	1181120
18	29	1562030
19	27	Run-out
20	23	Run-out
21	19	Run-out
22	15	Run-out

Table 2: Fatigue tensile results: stress amplitude, σ_a , and number of loading cycles to failure, N_f .

The experimental results exhibit a certain degree of scatter, particularly in the low/medium-cycle fatigue regime, reflecting the inherent variability associated with fatigue crack initiation and early crack propagation. Such scatter can be attributed to microstructural heterogeneities within the nanocomposite, including grain morphology induced by rolling, the distribution of MgZn₂ precipitates, and local variations in ZrO₂ nanoparticle dispersion, which act as preferential sites for fatigue crack initiation.

Considering that the tests are performed at a fatigue ratio $R = 0.10$, the Basquin relationship is applied in conjunction with the Morrow mean stress correction, according to:

$$\sigma_a = (\sigma'_f - \sigma_m) (2N_f)^b \quad (1)$$

where σ'_f is the fatigue strength coefficient, σ_m is the mean stress and b the Basquin's exponent.

To determine the material parameters σ'_f and b , a nonlinear least-squares fitting procedure is adopted. By taking the logarithm of the Morrow-corrected equation (Eqn.(1)), the problem is reformulated as:

$$\log \left(\frac{\sigma_a}{\sigma'_f - \sigma_m} \right) = b \cdot \log(2N_f) \quad (2)$$

For each trial value of σ'_f , the exponent b is estimated through linear regression in the $\log(2N_f)$ domain, and the corresponding fitting error is evaluated.

The parameter σ'_f is explored within a range of 100 – 580 MPa using a step of 10 MPa. To exclude oligocyclic fatigue effects, only experimental data corresponding to $2N_f > 5(10)^4$ are considered in the fitting procedure. The optimal parameter set is identified by minimising the least-squares error between experimental and predicted stress amplitudes. The minimum error, equal to 8 MPa, is obtained for $b = -0.2004$ and $\sigma'_f = 580$ MPa.

The S-N curve is reported in Fig. 7, along with the 10% – 90% scatter band. Using this curve, the stress amplitude corresponding to $N_f = 2(10)^6$ number of loading cycles is calculated by accounting for the mean stress effect at $R = 0.10$, yielding $\sigma_a = 26$ MPa. This relatively low fatigue strength may be attributed to the non-metallic inclusions observed in Fig. 1(b), which can act as local stress concentrators and preferential sites for fatigue-crack initiation, thereby reducing the fatigue life of the nanocomposite.

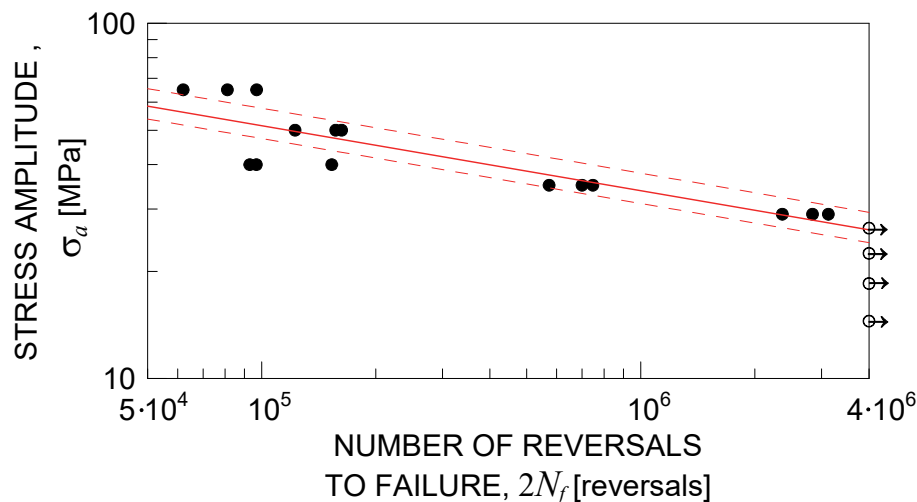


Figure 7: Stress amplitude, σ_a , versus the number of reversals to failure, $2N_f$, at $R = 0.10$. The solid line represents the Morrow-corrected Basquin fit, whereas the dashed lines delimit the 10% - 90% scatter band.

This interpretation is supported by the fracture surface analysis. As shown in Fig. 8, for specimen No. 18 (see Tab. 2), tested at a stress amplitude of 29 MPa, the fatigue crack appears to have initiated from a localised region close to the specimen surface, highlighted in the low-magnification SEM image and further detailed at higher magnification. The location of this region at the specimen edge, together with the apparent development of the fracture surface away from it, indicates a preferential crack-initiation site. This behaviour may be related to local stress concentration effects associated with microstructural heterogeneities, such as non-metallic inclusions, as previously observed in Fig. 1(b). Similar initiation features are also observed in the other tested specimens, suggesting that fatigue crack initiation is generally promoted by local microstructural heterogeneities.

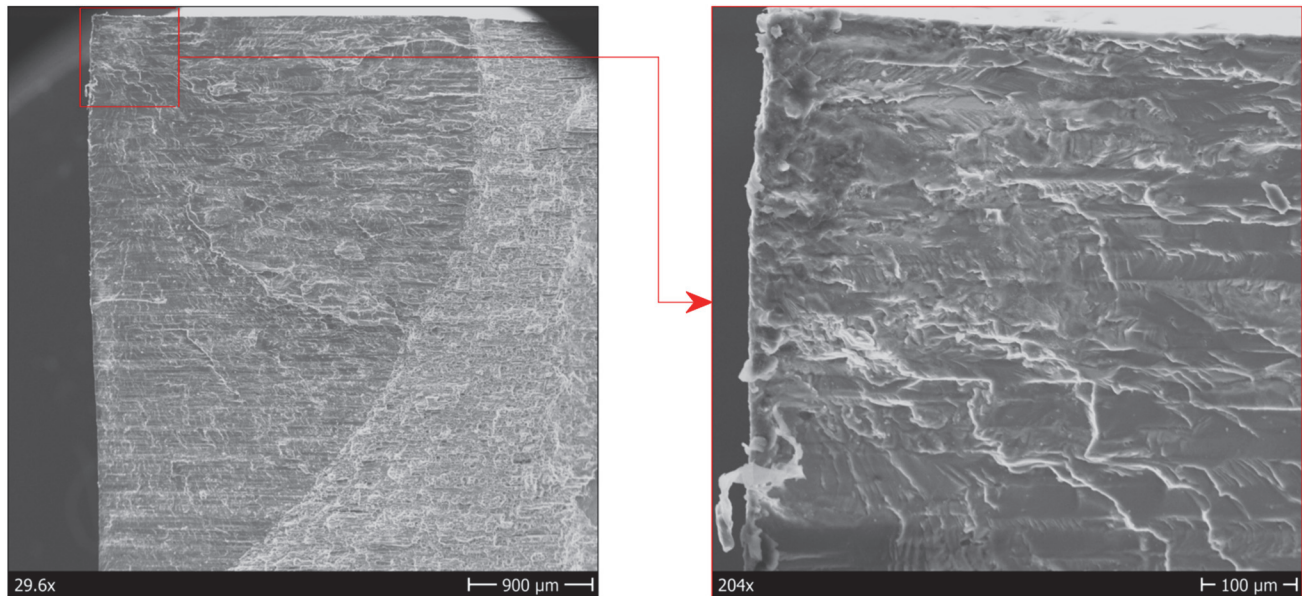


Figure 8: SEM analysis of the fracture surface of specimen No. 18, tested at a stress amplitude of 29 MPa: low-magnification image showing the overview of the fracture surface and the localised near-surface region where fatigue crack initiation is assumed to have occurred; high-magnification image providing a detailed view of the identified initiation region.

Tests on unreinforced specimens have not been carried out in this study, since the fatigue behaviour of Al7075 has been extensively investigated in the literature. The fatigue response is compared with that of the unreinforced Al7075-T6 alloy in the “Fatigue Behaviour” section.

Fatigue crack propagation behaviour

The fatigue crack propagation rate curves obtained from the three replicate tests performed for each fatigue ratio have shown negligible scatter, as illustrated, for example, for $R = 0.50$ in Fig. 9(a), where the crack growth rate, da/dN , is reported against the Stress-Intensity Factor (SIF) range, ΔK . Therefore, for clarity, only one representative curve for each fatigue ratio is reported in Fig. 9(b).

Usually, three distinct stages can be identified in the $da/dN - \Delta K$ diagram.

- Stage I (threshold stage): the crack growth rate is extremely low as ΔK approaches the threshold SIF range, ΔK_{th} . An accurate determination of ΔK_{th} , for a given fatigue ratio, requires specific experimental procedures, such as the load-shedding technique.
- Stage II (Paris stage): in the bilogarithmic $da/dN - \Delta K$ representation, a linear relationship is observed, which can be effectively described by the Paris law and provides a reliable interpolation of the experimental data.
- Stage III (final fracture stage): a rapid acceleration of the crack growth rate occurs, leading to unstable crack propagation and eventual specimen failure. This regime follows the end of the Paris region and is typically reached within a limited number of loading cycles.

With reference to Stage I, Fig. 9(b) shows that, for $R = 0.75$, a threshold SIF range of approximately $2.2 \text{ MPa}\sqrt{\text{m}}^{0.5}$ can be estimated.

With reference to Stage III, a marked acceleration of the crack growth rate da/dN is observed in Fig. 9(b) exclusively for $R = 0.50$, culminating in a final SIF range of approximately $13 - 14 \text{ MPam}^{0.5}$, corresponding to a fracture toughness of $26 - 28 \text{ MPam}^{0.5}$.

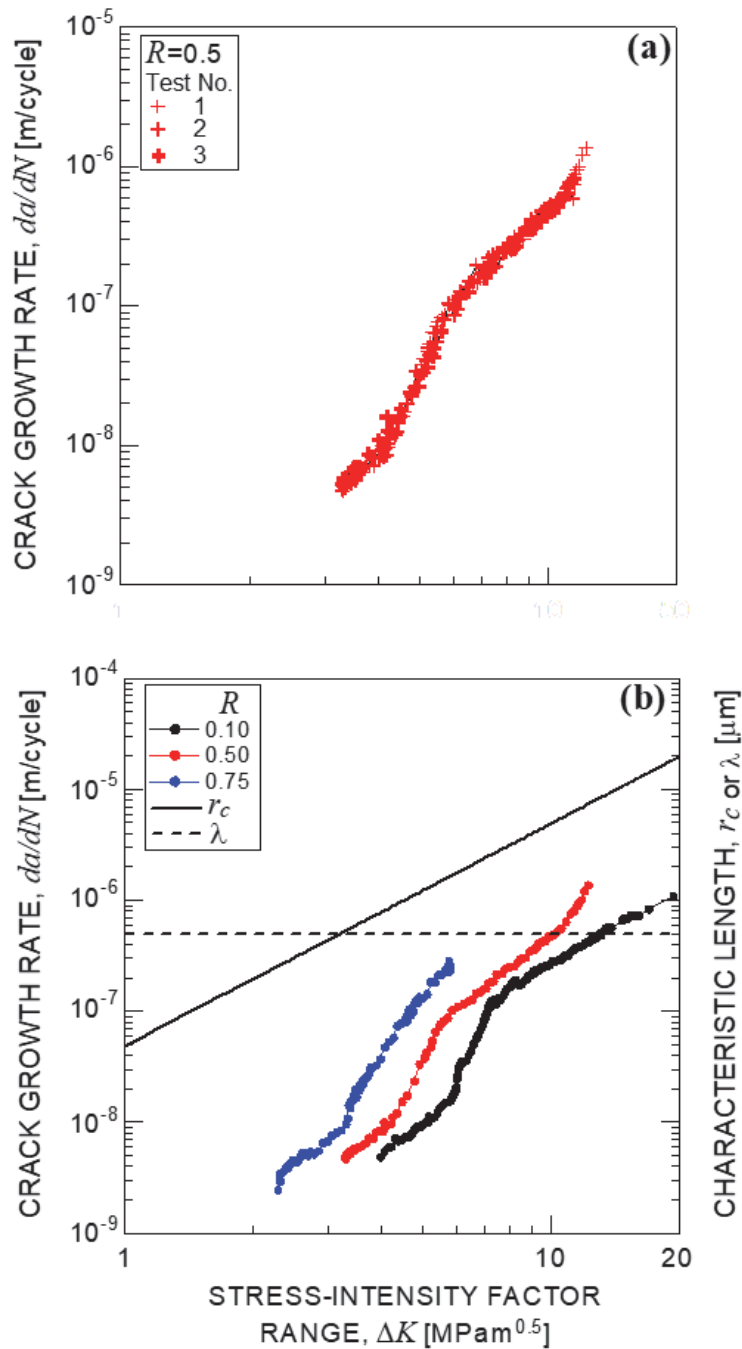


Figure 9: Fatigue crack propagation rate curves, da/dN vs. ΔK : (a) results obtained from the three replicate tests performed at $R = 0.50$ and (b) representative curves for the three investigated fatigue ratios ($R = 0.10, 0.50, 0.75$). The relationships between the characteristic lengths, namely cyclic plastic radius, r_c , and striation spacing, λ , and the applied SIF range, ΔK , are also reported.

With reference to Stage II, instead, the crack propagation behaviour can be further subdivided into distinct sub-stages, hereafter referred to as Sub-Stages IIa (immediately following Stage I), IIb (intermediate between IIa and IIc), and IIc (immediately preceding Stage III). For $R = 0.10$ and 0.50 , three sub-stages can be identified, each characterized by a

different slope in the bilogarithmic $da/dN - \Delta K$ representation. In contrast, for $R = 0.75$, only two sub-stages are observed, with Sub-Stage IIb being almost negligible.

By applying the Paris law ($da/dN = C\Delta K^m$) to each identified sub-stage, the corresponding parameters, namely the exponent m and the coefficient $\log C$ are determined. The results are summarised in Fig. 10 using a representation commonly adopted in the literature, where $\log C$ is plotted as a function of m , thus enabling a direct comparison among the different fatigue ratios and crack-growth sub-stages.

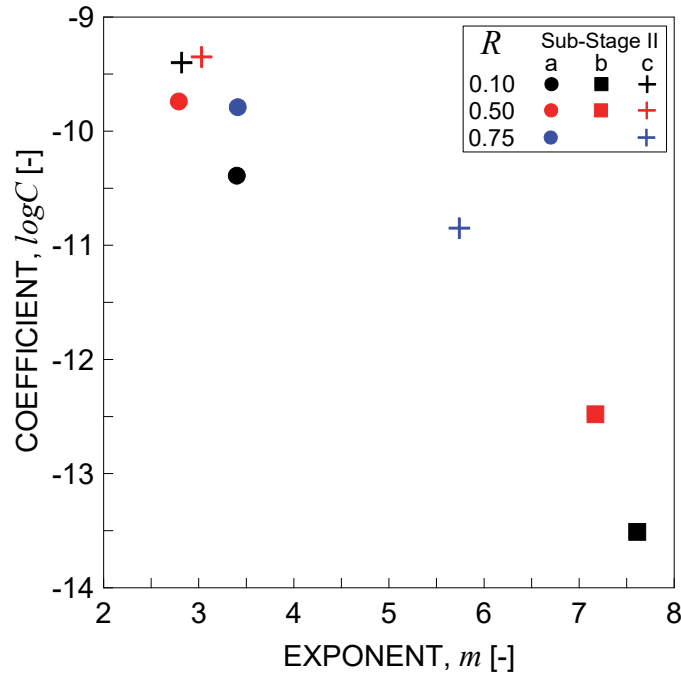


Figure 10: Values of $\log C$ and m for the sub-stages identified at the three investigated fatigue ratios.

The transition points $(\Delta K, da/dN)$, defined as the coordinates where the regression lines of two adjacent sub-stages intersect, are reported in Tab. 3 for each examined R value.

R	SUB-STAGE II					
	From a to b		From b to c		From a to c	
	ΔK [MPam ^{0.5}]	da/dN [m/cycle]	ΔK [MPam ^{0.5}]	da/dN [m/cycle]	ΔK [MPam ^{0.5}]	da/dN [m/cycle]
0.10	5.85	$1.66 (10)^{-8}$	7.22	$1.22 (10)^{-7}$	-	-
0.50	4.13	$9.46 (10)^{-9}$	5.62	$8.22 (10)^{-8}$	-	-
0.75	-	-	-	-	3.30	$9.56 (10)^{-9}$

Table 3: Data points $(\Delta K, da/dN)$ marking the transition between adjacent sub-stages for $R = 0.10, 0.50$, and 0.75 .

The occurrence of sub-stages characterised by different slopes within the Paris regime of fatigue crack propagation - commonly referred to as multilinear behaviour - has been widely reported in conventional Al alloys subjected to various heat treatments [36-38]. This behaviour is primarily attributed to changes in the dominant damage mechanisms, as well as to the interaction between the crack tip plastic zone and the underlying microstructural features. Three main mechanisms can be identified, as summarized below.



One important mechanism is the transition in fracture behaviour from brittle to ductile with increasing SIF range, as reported in Ref. [38]. At low load ranges, corresponding to low ΔK values (Sub-Stage IIa), the cyclic plastic zone size at the crack tip is smaller than the material grain size. As a result, cyclic plastic deformation cannot dominate, and crack growth occurs through a brittle mechanism, namely crystallographic fracture or cleavage. As ΔK increases toward intermediate levels (Sub-Stage IIb), a transition regime is observed, in which the fracture path becomes irregular and wavy, reflecting a mixture of cleavage features from the lower regime and emerging ductile features from the upper regime, without a single dominant damage mechanism. Finally, at high ΔK (Sub-Stage IIc), once the cyclic plastic zone becomes sufficiently large, cyclic plastic deformation becomes the dominant damage mechanism, and the fracture surface becomes less irregular, showing extensive and well-developed ductile striations associated with alternating slip ahead of the crack tip.

Another mechanism is the interaction between the crack tip plastic zone and microstructural barriers [38]. It is proposed that the multilinear behaviour is closely related to the interaction between the crack tip plastic zone and the physical obstacles present in the alloy microstructure. Changes in the slope of the $da/dN - \Delta K$ curve occur when the dimensions of the plastic zone - cyclic or monotonic - become comparable to characteristic microstructural features, which act as barriers to slip band transmission. For instance, transitions may occur when the cyclic plastic zone size approaches the average spacing between particles. The transition to the fully ductile regime (Sub-Stage IIc) is observed when the cyclic plastic zone becomes comparable to or larger than the grain size, allowing cyclic plastic deformation to fully develop and leading to the formation of well-defined fatigue striations.

In addition, the behaviour at higher crack growth rates is influenced by constraint loss associated with stress state transition [37]. At higher crack growth rates, the multilinear behaviour is further influenced by a macroscopic transition in the fracture plane, commonly referred to as the flat-to-slant transition. This transition is associated with a loss of constraint, corresponding to a shift from a highly constrained plane-strain condition to a plane-stress state. In the former, crack growth occurs on a flat fracture surface, whereas in the latter it progresses along a slanted path. This change in stress state is reflected in a modification of the crack growth behaviour, leading to a variation in the slope of the $da/dN - \Delta K$ curve and significantly affecting the effective SIF.

In the AMC investigated, ZrO_2 nanoparticles act similarly to intermetallic phases in conventional alloys [36-38], governing the interaction between crack tip plastic deformation and microstructural features. Ahead of the crack tip, two different plastic zones have to be considered in the discussion of fatigue crack propagation micromechanisms. The first is the monotonic plastic zone (identified by the radius, r_p), where the nanocomposite undergoes plastic deformation corresponding to the maximum value of the stress intensity factor, K_{max} :

$$r_p = \frac{1}{\alpha\pi} \left(\frac{K_{max}}{\sigma_y} \right)^2 \quad (3)$$

being α equal to 2 under plane stress conditions and to 6 under plane strain conditions, and σ_y is the yielding stress.

The second is the cyclic plastic zone (identified by the radius, r_c), defined as the region within the monotonic plastic zone that is subjected to repeated plastic deformation during each loading cycle. This represents the region where fatigue damage accumulates:

$$r_c = \frac{1}{\alpha\pi} \left(\frac{\Delta K}{2\sigma_y} \right)^2 \quad (4)$$

Based on the definitions of the monotonic and cyclic plastic zones, the corresponding values of ΔK , r_p , and r_c associated with the different sub-stage transitions are summarized in Tab. 4.

As shown in Tab. 4, the transition between sub-stages is associated with a progressive increase in both the monotonic and cyclic plastic zone sizes. In particular, the transition from Sub-Stage IIa to Sub-Stage IIc corresponds to significantly larger plastic zones, consistent with the development of fully ductile crack growth mechanisms. This trend is in agreement with the previously discussed mechanisms: the increase in plastic zone size promotes the transition from cleavage-dominated to ductile fracture behaviour, enhances the interaction between the crack tip plastic deformation and microstructural barriers

as their characteristic dimensions are approached, and contributes to the reduction of constraint associated with the transition from plane-strain to plane-stress conditions.

R	SUB-STAGE II								
	From a to b			From b to c			From a to c		
	ΔK [MPa $m^{0.5}$]	r_p [μm]	r_c [μm]	ΔK [MPa $m^{0.5}$]	r_p [μm]	r_c [μm]	ΔK [MPa $m^{0.5}$]	r_p [μm]	r_c [μm]
0.10	5.85	8.3	1.6	7.22	12.6	2.5	-	-	-
0.50	4.13	13.3	0.8	5.62	24.7	1.5	-	-	-
0.75	-	-	-	-	-	-	3.30	34.1	0.5

Table 4: Values of SIF range, monotonic and cyclic plastic zone sizes associated with the transitions between fatigue crack propagation sub-stages (IIa, IIb, and IIc), for $R=0.10$, 0.50 , and 0.75 .

The three sub-stages are also clearly identified through macrographs of the fracture surfaces (Fig. 11). In order to enhance the visual contrast between different regions, the original images are processed and converted into 40-level grayscale images. This procedure allows subtle variations in surface appearance to be highlighted, making the transitions between sub-stages more evident.

For $R=0.10$ (Fig. 11(a)) and $R=0.50$ (Fig. 11(b)), three distinct regions can be observed along the crack propagation direction, each characterised by a different grayscale level. These variations are consistent with the previously discussed mechanisms: the transition from brittle to ductile fracture behaviour, the increasing interaction between the crack tip plastic zone and microstructural barriers, and the progressive evolution of crack tip conditions with increasing ΔK . For $R=0.75$ (Fig. 11(c)), only a single transition in grayscale is observed, corresponding to the direct transition from Sub-Stage IIa to IIc, indicating the absence of a well-defined intermediate regime and reflecting a more rapid evolution of the governing mechanisms.

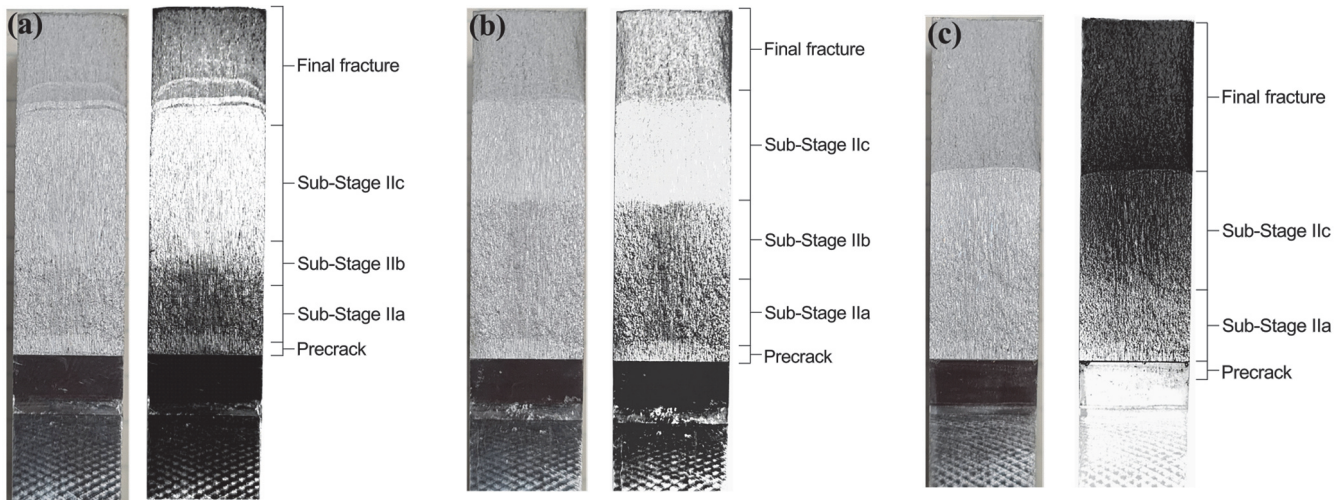


Figure 11: Macrographs of the fracture surfaces, processed in 40-level grayscale to enhance contrast, for different fatigue ratios, R : (a) 0.10 , (b) 0.50 and (c) 0.75 .

The ΔK_{th} values obtained from the two replicate tests performed for each fatigue ratio are listed in Tab. 5.

It can be observed that the results exhibit a high degree of repeatability. In addition, for $R=0.75$, the mean ΔK_{th} value obtained from the threshold tests is $2.24 \text{ MPa } m^{0.5}$, which is consistent with the approximate value of about $2.2 \text{ MPa } m^{0.5}$ extrapolated from the fatigue crack propagation curve reported in Fig. 9(b).

SEM fracture surface analysis of the specimens reveals different crack propagation morphologies, primarily depending on the propagation sub-stage (Figs. 12–14). In all micrographs, the crack propagation direction is horizontal, from left to right.

R	ΔK_{th}
	[MPa $\sqrt{\text{m}}^{0.5}$]
0.10	3.00
	3.22
0.50	2.84
	2.93
0.75	2.15
	2.32

Table 5: Threshold stress intensity factor range, ΔK_{th} , determined at different fatigue ratio, R , values.

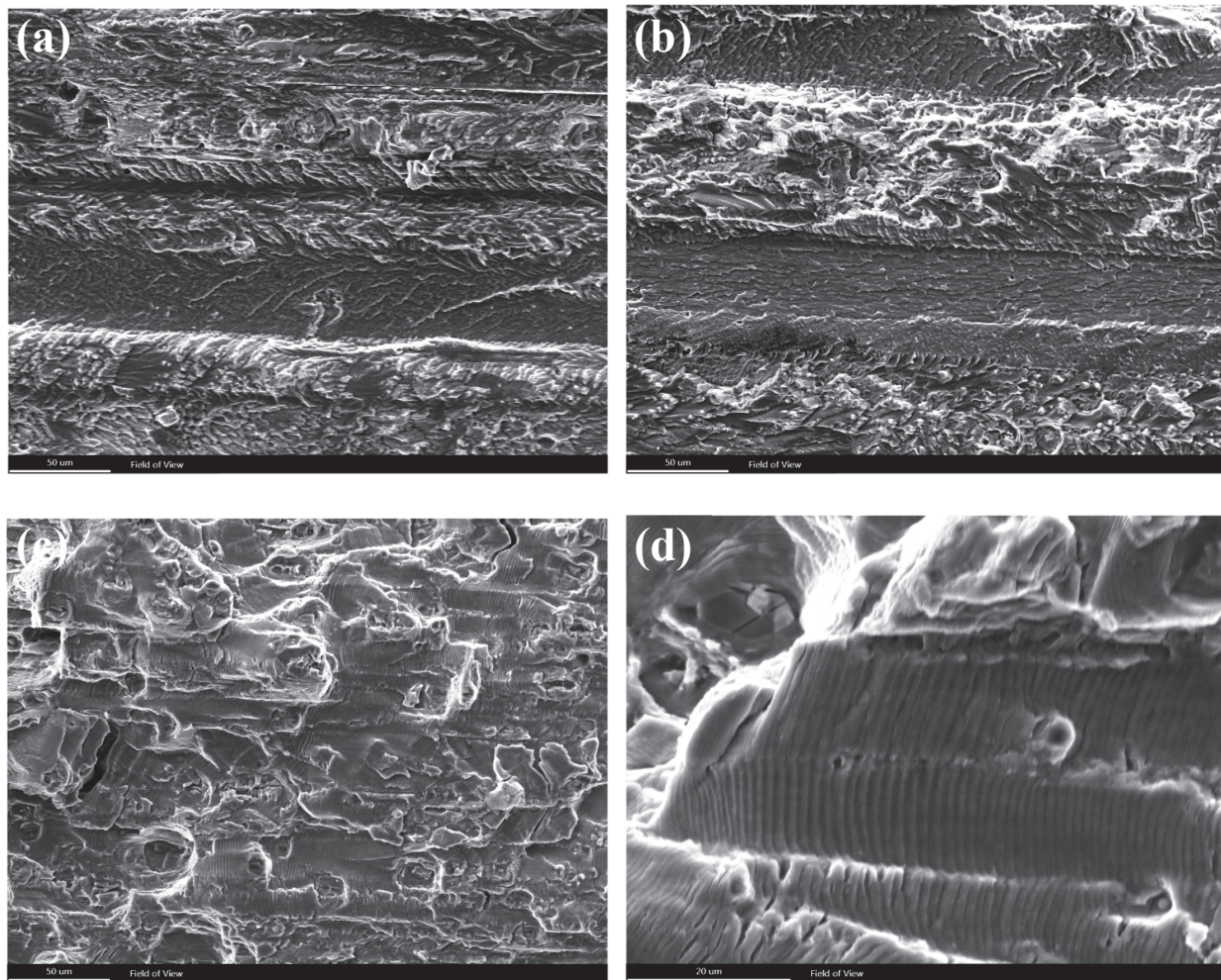


Figure 12: Fatigue fracture surface morphologies for $R = 0.10$: (a) Sub-Stage IIa, (b) Sub-Stage IIb, and (c) and (d) Sub-Stage IIc.

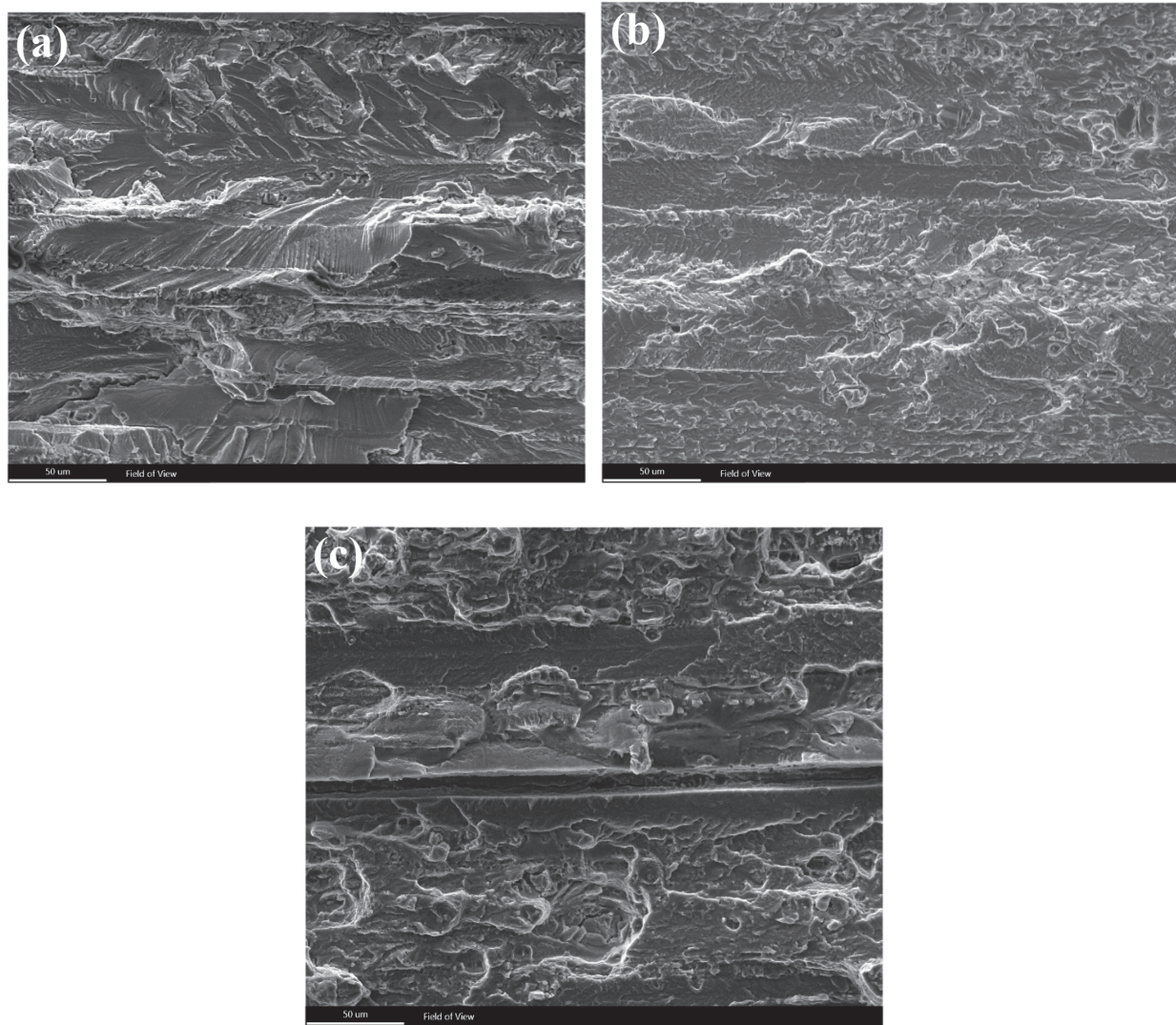


Figure 13: Fatigue fracture surface morphologies for $R = 0.50$: (a) Sub-Stage IIa, (b) Sub-Stage IIb, and (c) Sub-Stage IIc.

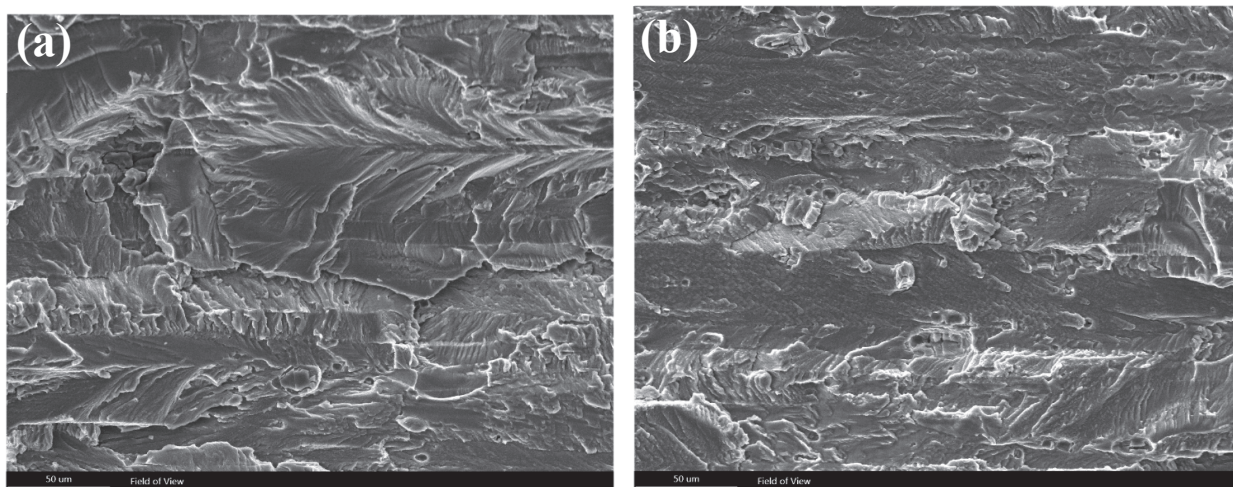


Figure 14: Fatigue fracture surface morphologies for $R = 0.75$: (a) Sub-Stage IIa, and (b) Sub-Stage IIc.



At lower ΔK values, corresponding to Sub-Stage IIa (see Figs. 12(a), 13(a), and 14(a)), fatigue striations are difficult to observe, and ZrO_2 nanoparticles act as the primary obstacles to crack propagation. At higher ΔK values (see Figs. 12(c) and (d), 13(c), and 14(b)), corresponding to Sub-Stage IIc, striations become more clearly visible; in addition, dimples are observed, likely due to the role of ZrO_2 nanoparticles as nucleation sites for microvoids. The larger extent of both the monotonic and cyclic zones at high ΔK allows the material to be treated more as a homogeneous medium than as a nanocomposite. For $R = 0.10$ and 0.50 , Sub-Stage IIb exhibits features characteristic of both adjacent sub-stages (see Figs. 12(b) and 13(b)), supporting its interpretation as a transition regime. In contrast, for $R = 0.75$, Sub-Stage IIb is not clearly observed, or can be considered negligible, as indicated by both the $da/dN - \Delta K$ curve and the fracture surface macrographs.

For all the investigated fatigue ratios, fatigue striations are consistently observed, with an approximately constant spacing, λ of about $0.5 \mu\text{m}$ (see Figs. 12(d), 13(a), and 14(a)). This suggests that, at lower ΔK values, the formation of a single striation requires several loading cycles. Conversely, at higher ΔK values (e.g., around $13 \text{ MPa m}^{0.5}$ for $R = 0.10$ and $10 \text{ MPa m}^{0.5}$ for $R = 0.50$, see Fig. 9(b)), each loading cycle approximately corresponds to a unit advance of the crack front. In some grains, crack propagation occurs transgranularly, and this becomes increasingly evident with increasing ΔK (e.g. Fig. 13). Moreover, at higher ΔK values, both intergranular and transgranular secondary cracks are observed (see Fig. 12(c), 13(c), and 14(b)).

Considering that: (i) the specimens are tested in the LT orientation, with crack propagation along the rolling direction (characterised by an average grain size of approximately $250 \mu\text{m}$); (ii) the mean striation spacing, λ , is approximately constant at about $0.5 \mu\text{m}$ for all the investigated R values and ΔK levels - although striations are difficult to observe at lower ΔK ; and (iii) the cyclic plastic zone radius, r_c , varies with the applied ΔK - as shown in Fig. 9(b) - it can be proposed that fatigue crack propagation in Stage II (multilinear regime) is governed by different mechanisms, as outlined below:

- for $R = 0.10$ and 0.50 , the influence of ZrO_2 nanoparticles is more pronounced at lower ΔK values. As ΔK increases, both the monotonic and cyclic plastic radii increase, leading to a condition in which the alloy can be treated as a homogeneous material rather than a nanocomposite. Consequently, three distinct sub-stages can be identified within the stable crack propagation regime: the lower sub-stage is dominated by the influence of ZrO_2 nanoparticles, whereas the upper sub-stage is governed by the intrinsic microstructural features of the nanocomposite, primarily the grain size;
- for $R = 0.75$, a different behaviour is observed, characterised by a negligible (or absent) Sub-Stage IIb. At the ΔK value corresponding to the change in slope of the $da/dN - \Delta K$ curve, the calculated cyclic plastic radius equals the measured striation spacing (see Fig. 9(b)). As a result, a gradual transition between Sub-Stages IIa and IIc, and the associated propagation mechanisms, is not observed. Therefore, for this fatigue ratio, only two sub-stages can be identified.

COMPARISON WITH DATA AVAILABLE IN THE LITERATURE

Static behaviour

The static mechanical properties measured in this study are compared with experimental data reported in the literature. However, most of the available studies failed to investigate the tensile behaviour of Al7075 reinforced with ZrO_2 nanoparticles. Since it is rather difficult to find data referring exactly to a nanocomposite with the same composition and processed by an identical technique as the one investigated in this study, the comparison is carried out using Al7075 matrix nanocomposites reinforced with zirconium dioxide nanoparticles, for which the average particle size differs by no more than $\pm 15\%$ and the reinforcement content is of the same order of magnitude as that analysed here ($1.5 \text{ wt}\%$), regardless of the fabrication process.

Experimental data reported by Sharma et al. [39] are analysed herein and used for comparison. The investigated AMC consisted of an Al7075 matrix reinforced with ZrO_2 nanoparticles, with an average particle size of 45 nm or less, and was fabricated using the powder metallurgy route. Four different nanoparticle volume fractions were considered, namely 1.25% , 2.53% , 3.84% and 5.19% , corresponding to 2.5% , 5.1% , 7.7% and 10.4% in weight fraction, respectively. Al7075 alloy without reinforcement was also used as a reference material. Tensile tests were performed using an Instron 8801 universal testing machine equipped with a 100 kN load cell, under a constant strain rate of 0.002 s^{-1} . For each nanoparticle content, three specimens were tested.



The results are reported in Tab. 6 in terms of yield stress, ultimate tensile strength, and fracture strength, together with the values obtained in the present study.

STUDY	ZrO ₂ [wt%]	ZrO ₂ size [nm]	$\sigma_{y0.2\%}$ [MPa]	σ_u [MPa]	σ_f [MPa]
Present	1.5	40	521	584	557
	0.0		337	506	506
	2.5		336	505	486
Ref. [39]	5.1	≤ 45	505	689	684
	7.7		479	648	649
	10.4		480	631	632

Table 6: Comparison of tensile properties: yield stress, $\sigma_{y0.2\%}$, ultimate tensile strength, σ_u , and fracture strength, σ_f , are reported for the present study and for literature data from Ref. [39], as a function of ZrO₂ nanoparticle content and average particle size.

For comparison purposes, particular attention is paid to the nanoparticle content closest to that investigated here, namely 2.5 wt% ZrO₂.

At comparable nanoparticle content, the yield stress obtained in the present study (521 MPa) is significantly higher than the value reported in Ref. [39] for 2.5 wt% ZrO₂ (336 MPa), corresponding to an increase of approximately +55%. A similar trend is observed for the ultimate tensile strength, which increases from 505 MPa to 584 MPa, yielding a difference of about +16%. The fracture strength also shows a marked improvement, rising from 486 MPa in Ref. [39] to 557 MPa in the present study, corresponding to an increase of approximately +15%. These discrepancies may be attributed to differences in processing routes, nanoparticle dispersion, interfacial bonding, and microstructural features, despite the comparable reinforcement content and particle size. In particular, the enhanced yield stress observed in the present study suggests a more effective activation of strengthening mechanisms, such as grain refinement and particle–dislocation interactions.

Moreover, compared to the unreinforced Al7075 alloy, the nanocomposite analysed in the present study shows a substantial enhancement in static mechanical properties. The addition of 1.5 wt% ZrO₂ nanoparticles results in an increase in the yield stress from 337 MPa to 521 MPa, corresponding to an improvement of approximately 55%. Similarly, the ultimate tensile strength increases from 506 MPa to 584 MPa, i.e., by about 15%. The fracture strength also improves from 506 MPa to 557 MPa, showing an increase of roughly 10%.

These quantitative improvements confirm the effectiveness of nanoscale ZrO₂ reinforcement in significantly enhancing the static load-bearing capability of the matrix even at low nanoparticle content.

Fatigue behaviour

To the best of the present authors knowledge, there is no information in the literature concerning cyclic tensile tests and fatigue crack propagation tests for the examined AMC. Consequently, the comparison reported below is carried out using literature data related to T6 heat-treated aluminum alloys, considering specimen thicknesses and fatigue ratios of the same order of magnitude as those investigated in the present study.

As far as cyclic tensile testing is concerned, the fatigue data reported by Oskouei and Ibrahim [40] for an Al7075-T6 alloy, tested in both uncoated and TiN-coated conditions, are analysed. Flat fatigue specimens with a thickness of 3.175 mm were machined from rolled plates according to ASTM E466, with their longitudinal axis aligned with the rolling direction. Fatigue tests were performed using a servo-hydraulic MTS 858 testing machine under constant-amplitude sinusoidal loading at a frequency of 15Hz and a fatigue ratio of $R = 0.10$. Considering only the results related to the uncoated specimens, for the above aluminum alloy, the number of reversals to failure, $2N_f$, is plotted against the applied stress amplitude, σ_a , in Fig. 15, along with the corresponding Morrow-corrected Basquin fit. The S–N curve of the nanocomposite is also shown for comparison.

The comparison shown in Fig. 15 highlights a significant reduction in fatigue strength for the AMC with respect to the conventional Al7075-T6 alloy over the entire investigated life range at $R = 0.10$. At a given number of reversals, the nanocomposite systematically exhibits lower stress amplitude values, indicating a detrimental effect of the reinforcement under cyclic loading. This behaviour can be interpreted in light of microstructural considerations, as the presence of second-phase particles may promote fatigue crack nucleation. In this regard, DeBartolo and Hillberry [41] demonstrated that, in several aircraft aluminum alloys including 7075-T6, fatigue cracks predominantly initiate at constituent particles, particularly when they are cracked or clustered, and that these features can be treated as intrinsic initial flaw sizes governing fatigue life.

The observed reduction in fatigue strength in the reinforced alloy may therefore be associated with an increased density of such microstructural heterogeneities or with an unfavourable particle distribution, which enhances the probability of crack initiation. Furthermore, the steeper slope of the S–N curve for the nanocomposite suggests a higher sensitivity to fatigue damage accumulation, while the larger scatter of the experimental data indicates a certain degree of microstructural variability, likely related to particle distribution and matrix–particle interactions.

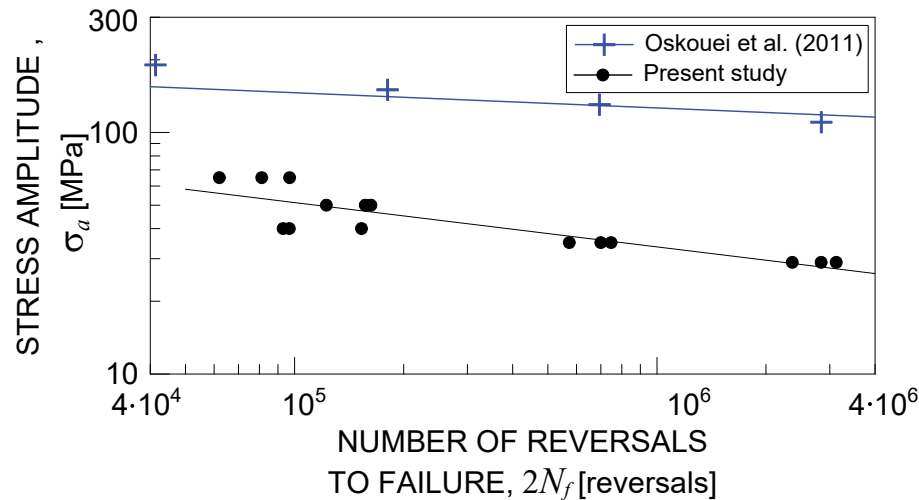


Figure 15: Stress amplitude, σ_a , as a function of the number of reversals to failure, $2N_f$, at $R = 0.10$, as reported in Ref. [40], where each data point represents the average number of reversals to failure at a given stress level. The blue solid line represents the Morrow-corrected Basquin fit. For comparison, the experimental data and fitted curve obtained in the present study (see Fig. 7) are also included.

As far as fatigue crack propagation testing is concerned, a comparison can be established between the investigated AMC and heat-treated aluminum alloys, based on the results reported by Newman and Walker [37] for an Al7075-T6 and by Borrego et al. [38] for an Al7050-T6.

In Newman and Walker [37], the fatigue crack propagation tests were performed on an Al7075-T6 aluminum alloy using both compact, C(T), and middle-crack tension, M(T), specimens machined from thin-sheet material with a thickness of 2mm. The specimen geometries were consistent with the ASTM E647 standard for fatigue crack propagation testing. The experiments were carried out under constant-amplitude loading conditions at fatigue ratios $R = 0.10$ and $R = 0.70$. Crack growth was monitored using compliance-based techniques, including back-face strain gauges and crack mouth opening displacement (CMOD) gauges, integrated within an automated crack monitoring system. The loading was applied using a servo-controlled fatigue testing system capable of maintaining the desired load history.

The $da/dN - \Delta K$ data for both this alloy and the examined nanocomposite are reported in Fig. 16, where only the curves obtained from C(T) specimens are shown.

Fig. 16 highlights a comparable fatigue crack propagation behaviour between the investigated AMC and the Al7075-T6 alloy reported in Ref. [37], although some differences can be observed depending on the fatigue ratio. At $R = 0.10$, the agreement between the AMC data and the literature trend appears to be particularly good, with the experimental points closely following the reference curve over a wide range of ΔK . This suggests that, under low mean stress conditions, the crack propagation mechanisms in the AMC are well described by those observed in the conventional Al7075-T6 alloy. Conversely, at higher fatigue ratios ($R = 0.70$ for Al7075-T6 and $R = 0.75$ for the AMC), although the overall trends remain similar, a slightly larger deviation from the reference curve can be observed. In this regime, the Al7075-T6 alloy exhibits a more pronounced multilinear behaviour with three distinct sub-stages in the Paris region, whereas the AMC displays a smoother response with only two sub-stages. This difference may be associated with the influence of the reinforcing phase, which can modify the crack propagation mechanisms and reduce the transitions between different propagation regimes, particularly at higher mean stress levels.

Borrego et al. [38] carried out fatigue crack propagation tests on an Al7050-T6 alloy, obtained through a T6 heat treatment involving solution treatment, quenching, and ageing. The experiments were performed using middle-cracked tension, M(T), specimens with a thickness of 6 mm, extracted from rolled plates in the longitudinal-transverse direction. The specimen geometry and testing procedures complied with the ASTM E647 standard for fatigue crack growth measurements. The tests were conducted under constant-amplitude loading using an Instron servo-hydraulic, closed-loop testing machine with a

capacity of 100kN, equipped with hydraulic grips and a computer-controlled data acquisition system. Crack length measurements were performed using a travelling microscope with a resolution of 10 μm . The tests were carried out over a range of fatigue ratios, $-1.0 \leq R \leq 0.5$. The loading frequency adopted for the fatigue tests was 25 Hz, with reduced frequency (0.5 Hz) during specific acquisition cycles for detailed load–displacement measurements.

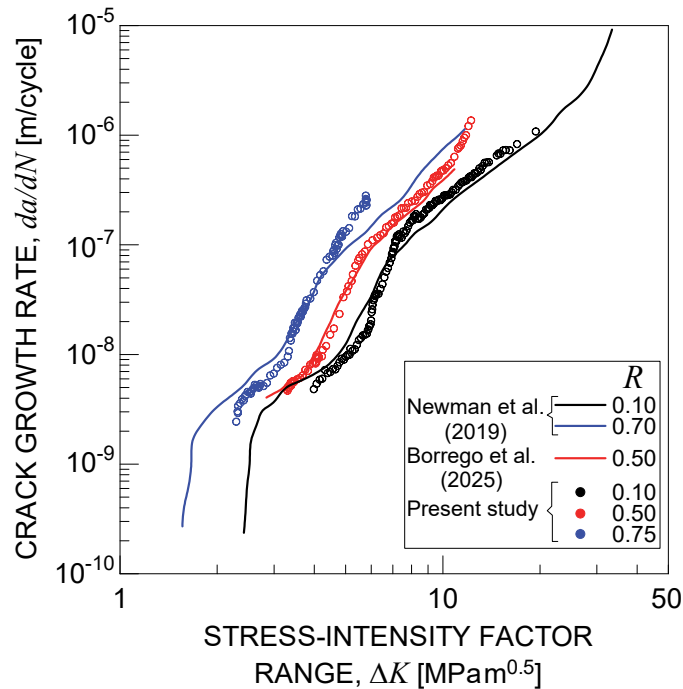


Figure 16: Fatigue crack propagation rate curves (da/dN vs. ΔK) at $R = 0.10$ and 0.70 , as reported in Ref. [37], and $R = 0.50$ as reported in Ref. [38], where only the trend lines are shown, rather than the original experimental points. For comparison, the experimental data obtained in the present study (see Fig. 9) are also included.

The da/dN - ΔK data for both this alloy and the examined nanocomposite are reported in Fig. 16, where only the curve obtained for $R = 0.50$ is shown.

The comparison between the red curve, corresponding to the Al7050-T6 alloy reported in Ref. [38], and the red experimental points of the present study reveals a very consistent fatigue crack propagation behaviour. In particular, both datasets exhibit a clear multilinear trend within the Paris regime, characterised by two distinct transition regions (from Sub-Stage IIa to Sub-Stage IIb and from Sub-Stage IIb to Sub-Stage IIc), which occur at comparable ΔK and da/dN values. The experimental points closely follow the literature trend over the entire investigated range, indicating that the crack propagation mechanisms governing the AMC are strongly aligned with those of the Al7050-T6 alloy. This agreement suggests that, despite the presence of reinforcement, the dominant fatigue crack growth processes remain similar, and the transitions between different propagation regimes are not significantly altered. Minor deviations observed at lower ΔK values may be attributed to microstructural differences or experimental scatter, but they do not affect the overall consistency between the two datasets.

Based on these experimental results [37, 38], it can be concluded that ZrO_2 nanoparticles in the AMC play a role analogous to that of intermetallic phases in heat-treated aluminum alloys, contributing to an enhanced resistance to fatigue crack propagation.

CONCLUSIONS

In the present study the fatigue behaviour of an Al7075 matrix reinforced with 1.5 wt% ZrO_2 nanoparticles has been investigated through a comprehensive experimental campaign including static tensile tests, cyclic tensile tests and fatigue crack propagation analyses. Based on the obtained results, the following conclusions can be drawn:



- The investigated nanocomposite exhibits high static mechanical properties, with a yield stress of about 521 MPa and an ultimate tensile strength of about 584 MPa, confirming the effectiveness of ZrO_2 nanoparticles in strengthening the aluminum matrix;
- The S-N results show a noticeable reduction in fatigue strength compared to conventional Al7075-T6 alloys. SEM observations suggest that this behaviour may be related to local stress concentration effects promoted by microstructural heterogeneities, such as non-metallic inclusions, which can favour preferential fatigue crack initiation;
- The fatigue crack propagation behaviour is characterised by a multilinear trend in the Paris regime. For fatigue ratios $R = 0.10$ and $R = 0.50$, three distinct Sub-Stages (IIa, IIb, and IIc) are identified, while for $R = 0.75$, only two Sub-Stages (IIa, and IIc) are observed, indicating a reduced transition regime at higher mean stress levels;
- The transitions between the sub-stages are strongly correlated with the evolution of the monotonic and cyclic plastic zone sizes, as well as with the interaction between the crack tip plasticity and microstructural features;
- Fractographic analysis confirms the transition from brittle to ductile crack propagation mechanisms with increasing ΔK , highlighting the role of nanoparticles as both obstacles to crack growth at low ΔK and as nucleation sites for microvoids at higher ΔK ;
- A good agreement is observed between the crack propagation behaviour of the investigated AMC and that of conventional heat-treated aluminum alloys reported in the literature, suggesting that ZrO_2 nanoparticles play a role analogous to intermetallic phases in governing fatigue crack propagation mechanisms;
- The combined analysis of fatigue lifetime and crack propagation provides a comprehensive description of the damage evolution process, representing a valuable contribution for the calibration and validation of fatigue models for nano-reinforced aluminum alloys.

REFERENCES

- [1] Wu, Q., Xu, W., Zhang, L. (2021). Advances in machining of particulate-reinforced metal matrix composites. In: Advanced Machining and Finishing - Handbooks in Advanced Manufacturing, by Gupta K. and Pramanik A., Elsevier, pp. 45–75.
- [2] Alam, M.A., Ya, H.H., Sapuan, S.M., Mamat, O., Parveez, B., Yusuf, M., Masood, F., Ilyas, R.A. (2022). Recent Advancements in Advanced Composites for Aerospace Applications: A Review. In: Advanced Composites in Aerospace Engineering Applications, by Mazlan N., Sapuan S.M., and Ilyas R.A., Springer, pp. 319-339.
- [3] Sreenivasa Iyengar, S.R., Sethuramu, D., Ravikumar, M. (2023). Study on Micro-structure, Hardness and Optimisation of Wear Characteristics of Al6061/TiB2/CeO2 Hotrolled MMCs using Taguchi Method. *Fracture and Structural Integrity*, 17(65), pp. 178-193.
- [4] Kuldeep, B., Ravikumar, K.P., Guruprasad, B.S., Tasneem, H.R.A., Ashrith, H.S., Shree, N., Ali, M.M., Bashir, M.N., Ahamad, T. (2023). A novel composite connecting rod: Study on mechanical and dynamic behaviour through experimental and finite element approach. *Composites Part C: Open Access*, 12, 100413.
- [5] Hatti, G., Lakshmikanthan, A., Naveen, G.J. (2023). Microstructure Characterization, Mechanical and Wear Behavior of Silicon Carbide and Neem Leaf Powder Reinforced AL7075 Alloy hybrid MMC's. *Fracture and Structural Integrity* 17(65), pp. 88-99.
- [6] Prakash, T.B., Gangadharappa, M., Somashekar, S., Ravikumar, M. The Impact of nanoparticles (B_4C - Al_2O_3) on mechanical, wear, fracture behavior and machining properties of formwork grade Al7075 composites (2024). *Fracture and Structural Integrity*, 18(69), pp. 210-226.
- [7] Pooja, K., Tarannum, N., Chaudhary, P. (2025). Metal matrix composites: revolutionary materials for shaping the future. *Discover Materials*, 5, 35.
- [8] Srivastava, S., Tevatia, A. (2026). Thermodynamic analysis of irreversibility during fatigue crack propagation in particle reinforced metal matrix composites. *Engineering Fracture Mechanics*, 335; 111930.
- [9] Bhushan, R.K. (2020). Optimization of tool wear parameters in machining AA7075/SiC composite, *Composites Part C: Open Access*, 2, 100035.
- [10] Ramezani, A., Jamshidi, Aval, H., Jamaati, R. Functionally gradient AA7075- Al_2O_3 composites fabrication via deformation-driven metallurgy process (2025). *Composites Part C: Open Access*, 17, 100599.
- [11] Rangrej, S., Pandya, S., Menghani, J. (2021). Effects of reinforcement additions on properties of aluminium matrix composites - A review. *Materials Today: Proceedings*, 44, pp. 637–641.



- [12] Dhongade, N.S., Meti, V.K.V., Siddhalingeshwar, I.G., Raju, G.U., Umarfarooq, M.A., Banapurmath, N.R., Sajjan, A.M., Uppin, V.S., Singh, B. (2025). Optimizing mechanical properties of AA7075 Metal Matrix Composites reinforced with TiB₂ and ZrO₂ particulates. *Fracture and Structural Integrity*, 19(74), pp. 1-19.
- [13] Casati, R., Vedani, M. (2014). Metal matrix composites reinforced by Nano-Particles—A review. *Metals*, 4, pp. 65-83.
- [14] Mahan, H.M., Konovalov, S.V., Najm, S.M., Mihaela, O., Trzepieciński, T. (2024). Experimental and Numerical Investigations of the Fatigue Life of AA2024 Aluminium Alloy-Based Nanocomposite Reinforced by TiO₂ Nanoparticles Under the Effect of Heat Treatment. *International Journal of Precision Engineering and Manufacturing*, 25, pp. 141-153.
- [15] Suchendra, K.R., Sreenivasa Reddy, M., Ravikumar, M. (2023). Influence of quenching agents on mechanical, wear, and fracture characteristics of Al₂O₃ / MoS₂ reinforced Al-6061 hybrid Metal Matrix Composite (MMCs). *Frattura ed Integrità Strutturale*, 17(63), pp. 122–133.
- [16] Kumar, G.B.V., Panigrahy, P.P., Nithika, S., Pramod, R., Rao, C.S.P. (2019). Assessment of mechanical and tribological characteristics of Silicon Nitride reinforced aluminum metal matrix composites. *Composites Part B: Engineering*, 175, 107138.
- [17] Abd-Elaziem, W., Khedr, M., Elsheikh, A.H., Liu, J., Zeng, Y., Sebae, T.A., Abd El-Baky, M.A., Darwish, M.A., Daoush, W.M., Li, X. (2024). Influence of nanoparticles addition on the fatigue failure behavior of metal matrix composites: Comprehensive review. *Engineering Failure Analysis*, 155, 107751.
- [18] Pu, Q., Geng, J., Li, K., Luo, T., Li, Y., Xia, P., Wang, F., Chen, D., Wang, H., Wang, H. (2025). Microstructural insights into short fatigue crack growth in particle-reinforced Al-matrix composite sheet. *International Journal of Fatigue*, 193, 108787.
- [19] Malaki, M., Tehrani, A.F., Niroumand, B. (2020). Fatigue behavior of metal matrix nanocomposites. *Ceramics International*, 46, pp. 23326-23336.
- [20] Srivatsan, T.S., Al-Hajri, M., Hannon, W., Vasudevan, V.K. (2004). The strain amplitude-controlled cyclic fatigue, deformation and fracture behavior of 7034 aluminum alloy reinforced with silicon carbide particulates. *Materials Science and Engineering: A*, 379, pp. 181-196.
- [21] Xia, J., Lewandowski, J.J., Willard, M.A. (2020). Tension and fatigue behavior of Al-2124A/SiC-particulate metal matrix composites. *Materials Science and Engineering: A*, 770, 138518.
- [22] Ganesan, K., Somasundaram Marimuthu, G., Hansda, S., Kumar Ramesh, V., Mani, S., Thangapandi, B. (2025). Fatigue crack growth rate behaviour of aluminium matrix composites reinforced with hollow glass microsphere. *International Journal of Fatigue*, 190, 108628.
- [23] Agrawal, A.P., Srivastava, S.K. (2024). Investigations of fatigue crack growth rate behaviour and life prediction of Si₃N₄/TiB₂ reinforced hybrid metal matrix composites. *International Journal of Fatigue*, 186, 108373.
- [24] Ma, L., Luo, X., Wang, H., Hu, R., Yi, X., Zhou, C., Chen, W., Ran, G. (2024). High fatigue performance and microscopic mechanisms of in-situ TiB₂/7050Al composite at different temperatures. *International Journal of Fatigue*, 185, 108372.
- [25] Senthilkumar, R., Arunkumar, N., Manzoor Hussian, M. (2015). A comparative study on low cycle fatigue behaviour of nano and micro Al₂O₃ reinforced AA2014 particulate hybrid composites. *Results in Physics*, 5, pp. 273-280.
- [26] Ceschini, L., Dahle, A., Gupta, M., Jarfors, A.E.W., Jayalakshmi, S., Morri, A., Rotundo, F., Toschi, S., Singh, R.A. (2017). *Aluminum and Magnesium Metal Matrix Nanocomposites*. Singapore: Springer.
- [27] Divagar, S., Vigneshwar, M., Selvamani, S.T. (2016). Impacts of Nano Particles on Fatigue Strength of Aluminum Based Metal Matrix Composites for Aerospace. *Materials Today: Proceedings*, 3, pp. 3734-3739.
- [28] Jaber, M.H., Aziz, G.A., Mohammed, A.J., AL-AIKawi, H.J. (2020). Electrical conductivity, magnetic and fatigue properties of aluminum matrix composites reinforced with nano-titanium dioxide (TiO₂). *Nanocomposites*, 6, pp. 47-55.
- [29] Zhang, Z., Zhao, Y., Wang, C., Tao, R., Fang, Z., Sun, Y., Kai, X. (2020). High cycle fatigue behavior of in-situ ZrB₂/AA6111 composites. *Materials Science and Engineering: A*, 788, 139590.
- [30] ASTM E8/E8M-22, (2024). *Standard Test Methods for Tension Testing of Metallic Materials*, ASTM International.
- [31] ASTM E466-21, (2021). *Standard Practice for Conducting Force-Controlled Constant-Amplitude Axial Fatigue Tests of Metallic Materials*, ASTM International.
- [32] ASTM E647-23a, (2024). *Standard Test Method for Measurement of Fatigue Crack Growth Rates*, ASTM International.
- [33] Bellini, C., Scorza, D., Vantadori, S., Iacoviello, F., Di Cocco, V. (2024). Fatigue crack path of an Al-Zn alloy reinforced with dispersed zirconium dioxide nanoparticles. *Procedia Structural Integrity*, 66, pp. 518-524.
- [34] ASTM E407-07(2015)e1, (2023). *Standard Practice for Microetching Metals and Alloys*, ASTM International
- [35] ASTM E112-13(2021), (2024). *Standard Test Methods for Determining Average Grain Size*, ASTM International.



- [36] Bianchetti, C., Zheng, R., Chromik, R.R., Lévesque, M., Brochu, M. (2018). Comparison of small and long fatigue crack growth behavior in AA 7050-T7451. *Engineering Fracture Mechanics*, 202, pp. 20-32.
- [37] Newman, J.C., Jr., Walker, K.F. (2019). Fatigue-crack growth in two aluminum alloys and crack-closure analyses under constant-amplitude and spectrum loading. *Theoretical and Applied Fracture Mechanics*, 100, pp. 307-318.
- [38] Borrego, L.P., Jesus, J.S., Branco, R., Ferreira, J.A.M., Antunes, F.V., Neto, D.M., Sérgio, E.R. (2025). Mechanisms of fatigue crack growth in 7050-T6 aluminium alloy. *International Journal of Fatigue*, 194, 108830.
- [39] Sharma, V., Mallick, A., Gupta, M. (2023). Enhancing significantly the strength and tribological response of Al7075 alloy using nano-ZrO₂ reinforcement. *Journal of Materials Research and Technology*, 27, pp. 7280-7292.
- [40] Oskouei, R.H., Ibrahim, R.N. (2011). The effect of a heat treatment on improving the fatigue properties of aluminium alloy 7075-T6 coated with TiN by PVD. *Procedia Engineering*, 10, pp. 1936-1942.
- [41] DeBartolo, E.A., Hillberry, B.M. (2001). A model of initial flaw sizes in aluminum alloys. *International Journal of Fatigue*, 23 (SUPPL. 1), pp. 79-86.